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13. SUPPLEMENTARY NOTES						
14. ABSTRACT This project used inert-gas condensation (IGC) to fabricate model nanostructured systems with the goal of better understanding the mechanisms responsible for decreasing the coercivity in soft magnetic materials. A new model system, Gd-Fe, was developed and investigated using IGC and melt spinning techniques. Atomic-level structural data acquired with synchrotron techniques was correlated to the magnetic properties of the materials.						
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## Cluster-Assembled Soft Magnets for Power Electronics Applications

Diandra Leslie-Pelecky, University of Nebraska

**The objectives of this project** were: 1) to use nanocluster assembly to produce model soft magnetic materials with simpler chemical composition than existing materials and well-controlled nanostructure, and 2) to use these materials to improve understanding of the fundamental mechanisms responsible for the soft magnetic properties. The following sections summarize the primary results of this project.

**Personnel:** A postdoctoral researcher, Lanping Yue, left in the middle of the project when a permanent job as a facility manager became available. A graduate student, Debbie Williams, left graduate school for family reasons and now is a high-school physics teacher. The change in personnel greatly delayed progress. The project was continued by David Schmitter, a graduate student, who is responsible for the majority of these results. Mr. Schmitter will graduate in August. We anticipate that three papers, all of which will acknowledge grant support from ONR, will be submitted by the end of the year.

**Development and Construction of an Inert-Gas Condensation/Compaction Chamber:** The primary result of this grant was the design and construction of an inert-gas condensation deposition chamber. We can deposit transition-metal, rare-earth and alloy nanoparticles with mean grain size  $D$  from 5 – 50 nm.

Figure 1 shows a transmission electron micrograph of IGC-Fe nanoparticles. We optimized deposition conditions to be able to produce 10-40 mg/h of nanoparticles with a size dispersion  $\Delta D/D \sim 0.1-1.0$ . The integrated system includes *in-situ* compaction and a transfer chamber to move samples to an inert-gas atmosphere without exposure to air. We made and studied Ni, FeCo, Gd, GdN,  $Gd_{1-x}Fe_x$  and Tb clusters, and Gd:GdN compacts. We can vary the grain sizes from less than 5 nm to over 50 nm by a combination of changing the deposition conditions and post-deposition annealing. This is a powerful method for studying grain-size dependence in soft magnets because it can produce large enough quantities of materials for extensive structural characterization and magnetic measurements, while maintaining precise control over the nanostructures.

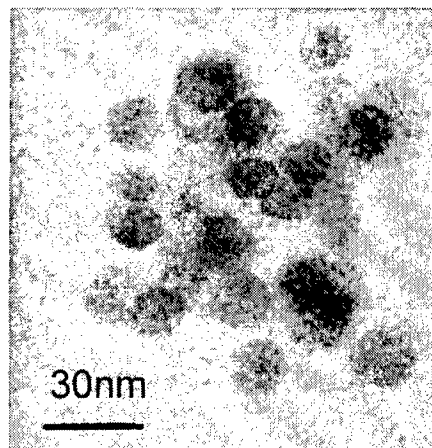


Figure 1: IGC-Fe nanoclusters.

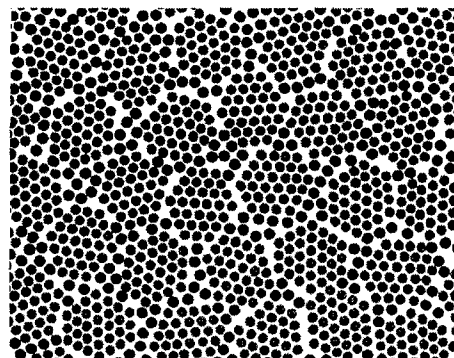


Figure 2: Cartoon representation of IGC-compacts nanostructure.

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### Exchange Coupling via Glassy Phases in Gd-Fe Nanostructures.

We studied Gd-based nanomaterials extensively to examine the mechanisms responsible for exchange coupling changes at the ferromagnetic transition. The 293 K room-temperature Curie temperature of Gd allowed us to track the magnetic behavior through the ferromagnetic transition without changing the nanostructure by applying high temperatures. The coercivity exhibits unexpected temperature dependence, as shown in Figure 3, including non-zero magnetization and coercivity on the order of 100 Oe well above the  $T_C$  of Gd.

Similar behavior was observed in GdN nanocompacts. GdN has a lower  $T_C$  and allows us to measure at temperatures far above  $T_C$ , again without changing the nanostructure. Figure 4 shows the temperature dependence of the zero-field-cooled magnetization (at 100 Oe) and the coercivity for an IGC-GdN sample with a grain size of 18 nm. (This sample was annealed at 600°C for 10 hours to complete the nitriding). Coercivity is observed in the nitrided sample at temperatures up to 400 K, which is significantly higher than the  $T_C$  of 60 K.

The cause of this unexpected behavior was difficult to identify. X-Ray diffraction, energy-dispersive x-ray spectroscopy, and electron diffraction did not indicate the presence of any secondary phases. It took Atomic Absorption Spectroscopy (AAS) to show that very small (0.5-2 wt. %) amounts of Fe were present in the samples. The culprit ultimately was identified as failure of the sputtering gun to confine the plasma to the target.

The decrease in the coercivity suggests that there are two phases. One is the Gd or Gd-N phase that orders at its expected  $T_C$ , although  $T_C$  may be depressed from the bulk value if the grain size is small. The second phase is ordered above  $T_C$ . When the Gd or GdN phase orders, anisotropy averaging decreases the coercivity. It remains unclear why very small amounts of iron have such a significant effect on the magnetic properties; however, the system allows us to investigate the behavior of the exchange averaging as the ferromagnetic temperature is reached.

To understand the mechanism, we fabricated Gd-Fe samples with Fe concentrations ranging from 1 wt. % to 40 wt. %. The intermediate part of this range has not been studied much because of the limited solubility of Fe in Gd. We made samples using IGC and melt spinning (another non-equilibrium fabrication technique) to overcome the limited solubility and understand how iron affects the magnetic properties. X-ray diffraction shows only peaks from hcp-Gd

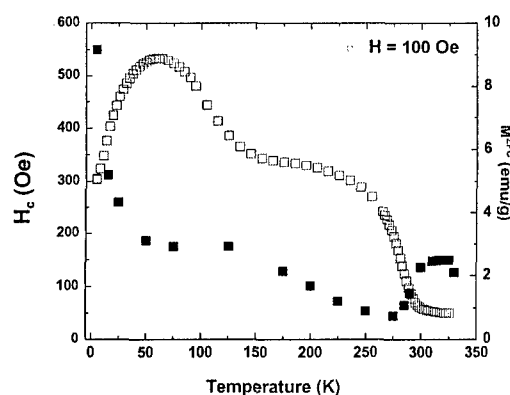


Figure 3: The zero-field-cooled magnetization measured at 100 Oe (open squares) and the coercivity (closed circles) as functions of temperature for an IGC-Gd compact.

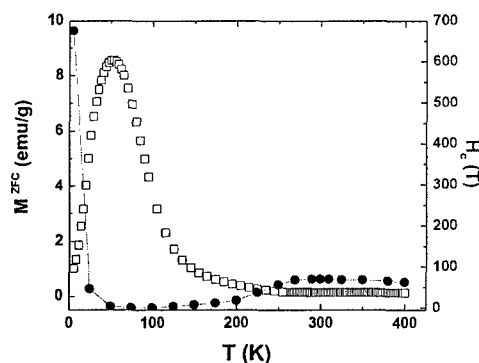


Figure 4: The zero-field-cooled magnetization measured at 100 Oe (open squares) and the coercivity (closed circles) as functions of temperature for an IGC-GdN compact.

crystallites in  $\text{Gd}_{1-x}\text{Fe}_x$  for  $x$  up to 0.70. A combination of x-ray diffraction, electron microscopy, and electron diffraction suggests that there is a glassy GdFe phase in which hcp Gd crystallites are embedded. The glassy Gd-Fe phase is magnetic at temperatures up to at least 400 K.

Determining the details of the nanostructure is critical to understanding the origin of the magnetic behavior. This is especially complex because the second phase is not evident in x-ray or electron diffraction. We applied for and received time to do XAFS at the Advanced Photon Source to investigate the nature of the second phase. This data are being analyzed, but some preliminary XAFS results are shown in Figure 5 for melt-spun  $\text{Gd}_{1-x}\text{Fe}_x$ . The data show significant changes in the positions of the Gd and Fe atoms as the composition is changed. These data are being fit using a combination of hcp-Gd crystallites and an amorphous phase, and

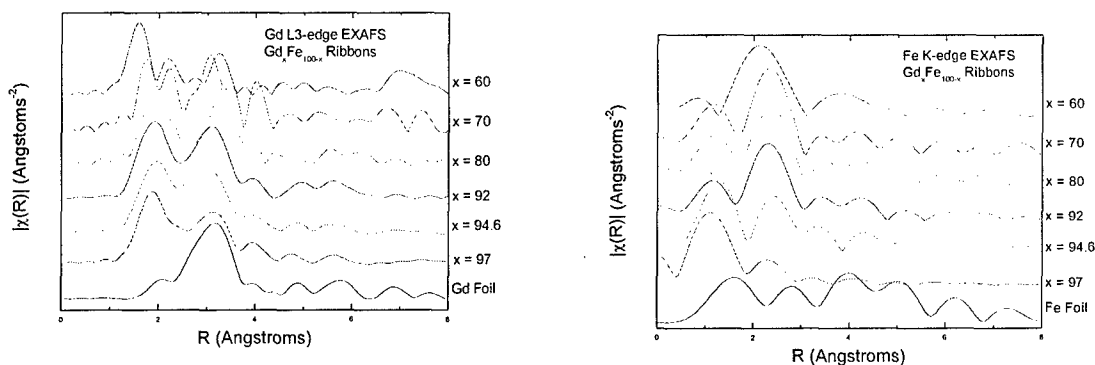


Figure 5: EXAFS data for  $\text{Gd}_{1-x}\text{Fe}_x$  on the Gd edge (left) and the Fe edge (right).

will be correlated to the magnetic behavior.

The coercivity is shown in Figure 6 for different values of  $x$ . Importantly, these data are taken at 310 K, above  $T_C$  of Gd. The shape of the loop also indicates the sensitivity of the magnetic properties to the presence of iron.

**Conclusions.** In addition to developing a fabrication system with the ability to control nanostructure across a wide range of lengths, we have identified a new model system for studying exchange coupling and its effect on coercivity. The  $\text{Gd}_{1-x}\text{Fe}_x$  system has a number of advantages over more complicated soft magnet materials. A particular strength of this as a model system is that the glassy phase appears to be fairly uniform, so modeling the behavior of that phase is significantly easier than in ternary or quaternary systems, where composition gradients can greatly change the magnetic properties on very short length scales.

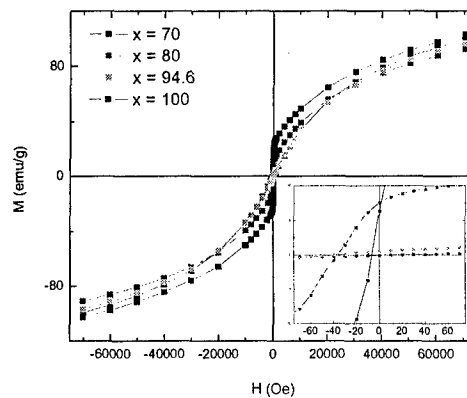


Figure 6: Magnetization vs. temperature at 310 K for  $\text{Gd}_{1-x}\text{Fe}_x$ .